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A Method for the Calibration of Class 2 and Class 4 Standards of Mass

By Nabil A. Bibawy

UNITED STATES DEPARTMENT OF THE INTERIOR

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(United States. Bureau of Mines)

Information Circular 9230

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UNITED STATES DEPARTMENT OF THE INTERIOR
Manuel Lujan, Jr., Secretary

BUREAU OF MINES
T S Ary, Director

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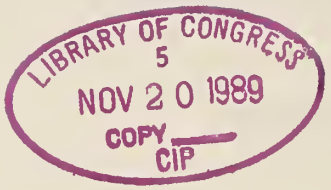

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

g	gram	mg	milligram
kg	kilogram	pct	percent
L	liter		

A METHOD FOR THE CALIBRATION OF CLASS 2 AND CLASS 4 STANDARDS OF MASS

By Nabil A. Bibawy[1]

ABSTRACT

The U.S. Bureau of Mines has established a method for the calibration of class 2 and class 4 standards of mass using a procedure from designs developed by the National Institute of Standards and Technology (NIST). This method produces a weight calibration that is within 1.0 pct of the balance sensitivity. A set of four 1-kg weights yielded a standard deviation of 3.25 mg compared to ±2.91 mg balance sensitivity. The method employs the use of a high-accuracy balance to measure the difference between a set of weights calibrated by NIST and the set to be calibrated. The error of the balance is included in the error for each weight calibrated.

[1]Chemist, Helium Field Operations, U.S. Bureau of Mines, Amarillo, TX.

INTRODUCTION

The U.S. Bureau of Mines investigates methods to improve helium analysis for the certification of calibration standards used in helium production, purification, liquification, transportation, and storage operations. Since the ultimate composition of primary standard gas mixtures depends upon the accuracy of the balance weights used, the weights require accurate calibration. Calibrating the weights against existing NIST certified weights provides the best solution because the error of the balance is included in the calibration.

The true mass of an object is usually determined on an analytical balance by comparing the object to a group of weights whose individual masses are accurately known. True mass of the balance weight is determined by comparing it to known standards of mass and applying the correction value to make the weight agree with the standards.

Preparation of primary standard gas mixtures for analytical instruments was described by Miller, Carroll, and Emerson in 1965.[2] The nominal mass of the balance weight is the quantity of weight actually observed on the balance from comparison to the weight(s) of a weight set and the balance chain scale. The correction value is added to or subtracted from the nominal weight to obtain the true mass.

Designs for the calibration of standards of mass were discussed by Cameron, Croarkin, and Raybold in 1977.[3] This collection of designs for the intercomparisons of sets of weights was used as a resource for determining the correction values for balance weights used in the preparation of primary standard gas mixtures. The designs used are A.1.2 for the calibration of four equal weights (appendix A), and C.10.5.2.2.1.1.1 for the calibration of weights less than 1 kg (appendix B), and larger than 1 kg (appendix C). These designs provide for a check standard weight, which is treated as an additional unknown weight and is used for monitoring the performance of the calibration process.

Because the weights calibrated in this study are compared against weights of the same metal, the buoyancy corrections are not required.

ACKNOWLEDGMENTS

The author thanks Dr. Richard S. Davis, physicist, Mass Division, Center for Basic Standards, National Institute of Standards and Technology, Gaithersburg, MD, for his technical assistance.

BALANCE AND WEIGHTS

The Voland model HCE 100G[4] analytical balance is a double-pan balance used for the comparison of weights against standards (fig. 1). The high precision and large capacity of this balance make it suitable for the preparation of weighed gaseous mixtures in standard-size cylinders. An analysis using a double-substitution procedure to determine the reproducibility of the Voland balance yielded a precision of ±2.91 mg.

Class 2 and class 4 sets of stainless steel weights with individual masses ranging from 1 g to 5 kg were supplied with the balance. These sets were calibrated using the procedures described in this report.

The weights used as the standard and check standard are of a class M set with 1-g to 1-kg masses of stainless steel, 30- to 500-mg masses of tantalum, and 1- to 20-mg masses of aluminum. This set was made by Wm. Ainsworth and Sons, Inc., and calibrated by NIST (table 1).

Table 1.–National Institute of Standards and Technology report of calibration for class M weights

Item	Mass Correction, mg		Uncertainty,	Volume at
	Apparent	True	mg	20° C, cm^3
1 kg	2.285	11.039	0.069	126.4716
500 g	1.722	6.292	.040	63.3962
300 g	.923	3.646	.034	38.0233
200 g	.627	2.443	.027	25.3489
100 g	-.3111	.6357	.0075	12.70656
50 g	.0665	.5205	.0056	6.33720
30 g	-.1964	.0761	.0058	3.80229
20 g	.0473	.2290	.0050	2.53488
10 g	.0166	.1074	.0064	1.26744
5 g	-.0239	.0215	.0048	.63372
3 g	-.0228	.0045	.0033	.38023
2 g	-.0184	-.0002	.0024	.25349
1 g	.0044	.0135	.0023	.12674
500 mg	-.0052	-.0406	.0017	.030118
300 mg	-.0063	-.0275	.0011	.018070
200 mg	-.00886	-.02300	.00083	.012046
100 mg	-.00425	-.01132	.00078	.006023
50 mg	-.00197	-.00551	.00055	.003011
30 mg	.00216	.00004	.00054	.001807
20 mg	-.00275	.00327	.00046	.007408
10 mg	.00384	.00686	.00059	.003706
5 mg	-.00434	-.00283	.00049	.001850
3 mg	.00424	.00515	.00052	.001113
2 mg	-.00204	-.00144	.00045	.000740
1 mg	.00353	.00384	.00059	.000371

[2]Miller, J. E., A. J. Carroll, and D. E. Emerson. Preparation of Primary Standard Gas Mixtures for Analytical Instruments. BuMines RI 6674, 1965, 10 pp.

[3]Cameron, J. M., M. C. Croarkin, and R. C. Raybold. Designs for the Calibration of Standards of Mass. NBS Tech Note 952, June 1977, 64 pp.; NTIS PB 268499.

[4]Reference to specific products does not imply endorsement by the U.S. Bureau of Mines.

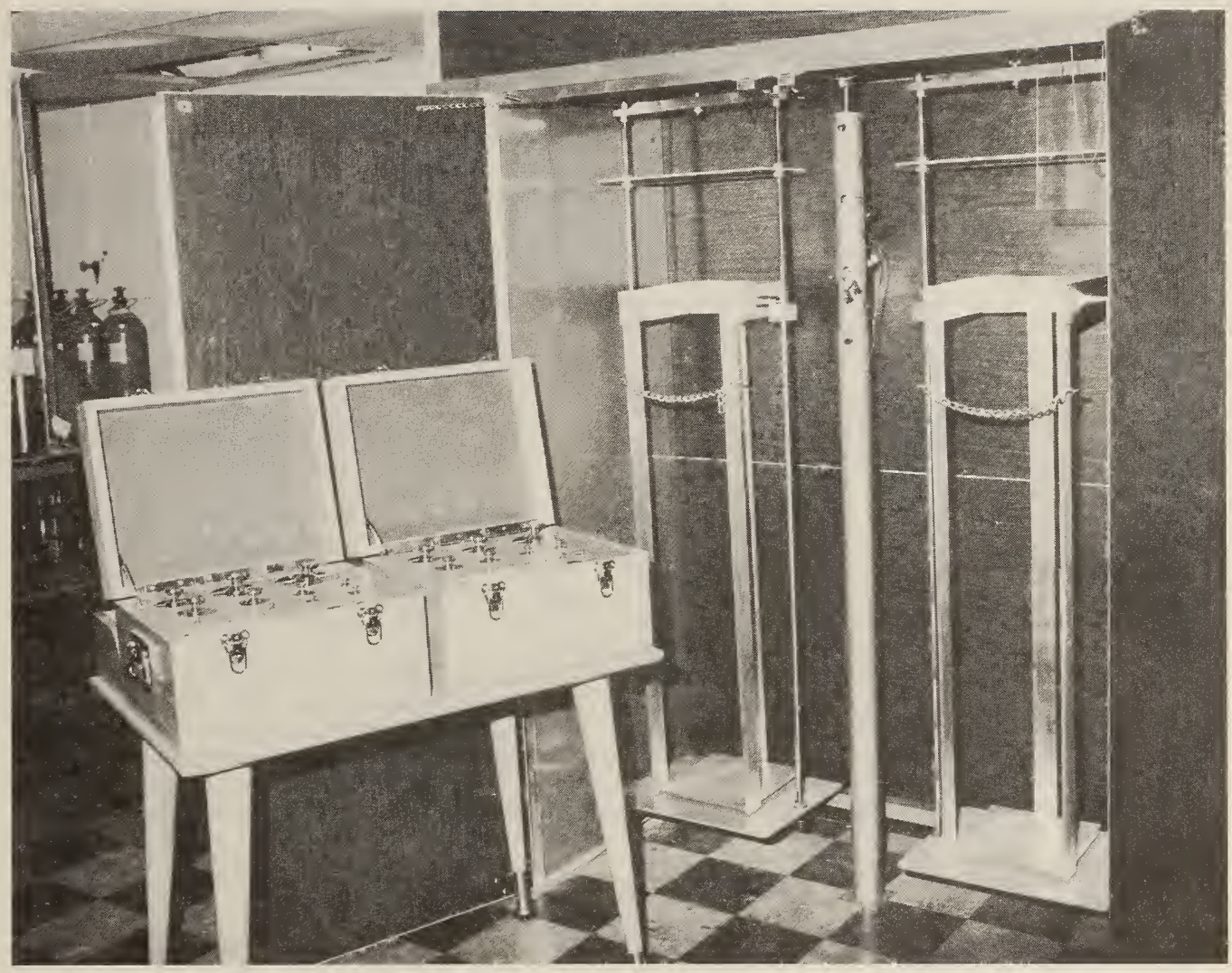

Figure 1.–Voland model HCE 100G balance, class 2 and class 4 sets of weights.

EQUATIONS AND DESIGNS FOR WEIGHING COMPARISONS

The equations and methods of comparing weights used in this report were performed in accordance with NBS Technical Note 952[5] and are called designs.

To calibrate a set of weights, multiple comparison must be made of one weight or set of weights against another.

Using the first comparison as an example, Y_1 is the weight that must be added to the pan carrying X_2 in order to balance X_1. If weight must be added to X_1 in order to balance X_2, then that weight with a negative sign becomes Y_1, indicating that X_1 is less than X_2.

For four equal weights, $X_{(1\ to\ 4)}$, six comparisons are made as follows:

$$
\begin{array}{lcl}
X_1 & \text{against} & X_2 = Y_1 \\
X_1 & \text{against} & X_3 = Y_2 \\
X_1 & \text{against} & X_4 = Y_3 \\
X_2 & \text{against} & X_3 = Y_4 \\
X_2 & \text{against} & X_4 = Y_5 \\
X_3 & \text{against} & X_4 = Y_6
\end{array}
$$

where $X_{(1\ to\ 4)}$ = weights to be compared

and $Y_{(1\ to\ 6)}$ = observed differences for the above comparisons.

[5]Work cited in footnote 3.

The correction values (B) for each weight (or combination of weights) were calculated as follows:

$$B_1 = [Y_1K_{(X_1,Y_1)} + Y_2K_{(X_1,Y_2)} + \cdots + M_{(X_1)}R]/D$$

$$B_2 = [Y_1K_{(X_2,Y_1)} + Y_2K_{(X_2,Y_2)} + \cdots + M_{(X_2)}R]/D$$

$$\vdots$$

$$B_n = [Y_1K_{(X_n,Y_1)} + Y_2K_{(X_n,Y_2)} + \cdots + M_{(X_n)}R]/D \quad (1)$$

where B_n = the calculated correction value for weight X_n, mg,

Y_n = the observed difference in the (n)th comparison being made, mg,

$K_{(X_n,Y_n)}$ = parameter value for the design being used,

$M_{(X_n)}$ = restraint value for the design being used,

R = the previously determined true mass correction value for the standard weight or combination of standard weights, mg,

and D = the divisor for the design being used.

The true mass for each weight (X) is determined by adding or subtracting the correction value B to the nominal value of the standard weights being compared.

The deviations (Dev) are found in a similar manner to the B values, using the coefficients from the same designs. The deviations are calculated as follows:

$$\text{Dev (1)} = [Y_1 L_{(Z_1,Y_1)} + Y_2L_{(Z_1,Y_2)} + \cdots]/D$$

$$\text{Dev (2)} = [Y_1 L_{(Z_2,Y_1)} + Y_2 L_{(Z_2,Y_2)} + \cdots]/D$$

$$\vdots$$

$$\text{Dev (n)} = [Y_1 L_{(Z_n,Y_1)} + Y_2 L_{(Z_n,Y_2)} + \cdots]/D \quad (2)$$

where Dev (n) = calculated deviation corresponding to observation Y_n, mg,

Y_n = the observed difference in the (n)th comparison, mg,

$L_{(Z_n,Y_n)}$ = the parameter value (deviation) from the design being used,

and D = the divisor for the design being used.

The standard deviation of the particular set of measurements is calculated as follows: The deviations are computed, squared, summed, and divided by the degrees of freedom for the particular design being used. The square root of this number is the standard deviation for the measurements. This should not be significantly different from the standard deviation of the balance of ±2.91 mg.

CALIBRATION PROCEDURE

CALIBRATION OF 1-kg WEIGHTS

NIST design A.1.2 (appendix A) was used in the calibration procedure for a 1-kg weight or a combination of weights equivalent to 1 kg. In the calibration procedure, a 1-kg weight (X_1) and a combination of weights equaling 1 kg (X_2) from the desired set were compared to an NIST-calibrated class M check standard (X_3) and standard (X_4). The check standard was a combination of weights equaling 1 kg, and the standard was a 1-kg weight.

CALIBRATION WITHIN A SET OF WEIGHTS

NIST design C.10.5.2.2.1.1.1 (appendix B) was used in the calibration procedure for weight sets with denominations of 500 g, 200 g, 200 g, and 100 g; 50 g, 20 g, 20 g, and 10 g; and 5 g, 2 g, 2 g, and 1 g; or other combinations with the ratio of 5:2:2:1. In the calibration procedure, a 500-g, 50-g, or 5-g weight was used as X_1, a 200-g, 20-g, or 2-g weight was used as X_2, a 200-g, 20-g, or 2-g weight was used as X_3, and a 100-g, 10-g, or 1-g weight was used as X_4. The desired set was compared to a check standard (X_5) comprised of a combination of 50-g, 20-g, 20g, and 10-g weights, 5-g, 2-g, 2-g, and 1-g weights, or 500-mg, 200-mg, 200-mg, and 100-mg weights.

Weight X_6 was the standard obtained from the NIST-calibrated class M set and was a 100-g, 10-g, or 1-g weight. Eight different comparisons were made on the Voland balance as follows:

X_1+X_6	against	$X_2+X_3+X_4+X_5$	$= Y_1$
X_1+X_5	against	$X_2+X_3+X_4+X_6$	$= Y_2$
X_1+X_4	against	$X_2+X_3+X_5+X_6$	$= Y_3$
X_1	against	$X_2+X_4+X_5+X_6$	$= Y_4$
X_1	against	$X_3+X_4+X_5+X_6$	$= Y_5$
X_2+X_4	against	X_3+X_5	$= Y_6$
X_2+X_6	against	X_3+X_4	$= Y_7$
X_2+X_5	against	X_3+X_6	$= Y_8$

The Y (1 to 8) values are the observations obtained for the above comparisons. The correction values (B) and deviations were calculated using equations 1 and 2, respectively, and the parameters and restraint from NIST design C.10.5.2.2.1.1.1 shown in appendix B. The standard deviation of a particular set of measurements was calculated using the method outlined previously.

To calibrate weights larger than 1 kg, the same NIST design (C.10.5.2.2.1.1.1) was used, but with a different restraint, divisor, and parameter values as shown in appendix C. Correction values, deviations, and the standard deviations were calculated using equations 1 and 2 and the same steps as outlined above.

CALIBRATION RESULTS

The calibration results of the class 2 and class 4 sets using the class M, NIST-calibrated set as standards are given in tables 2 and 3 respectively. The results of calibrations for individual weights follow.

Table 2.–Individual comparisons of class 2 weights with the class M standard

Weight	Item	Identification mark	True mass of, correction, mg	Standard deviation of correction, mg	Standard deviation for a set, mg
1	5 kg	None	93.052	7.06	3.62
2	2 kg	X	34.935	3.69	3.62
3	2 kg	None	18.507	3.69	3.62
4	1 kg	None	14.789	2.30	2.04
5	500 g	None	9.091	1.06	2.67
6	200 g	X	3.065	1.25	2.67
7	200 g	None	.208	1.25	2.67
8	100 g	None	3.675	1.41	2.67
9	50 g	None	2.909	1.31	2.44
10	20 g	X	-.122	1.29	2.44
11	20 g	None	-1.551	1.29	2.44
12	10 g	None	.296	1.42	2.44
13	5 g	None	1.577	1.31	2.18
14	2 g	None	2.059	1.29	2.18
15	2 g	X	-.798	1.29	2.18
16	1 g	None	-.399	1.42	2.18

Table 3.–Individual comparisons of class 4 weights with the class M standard

Weight	Item	Identification mark	True mass of, correction, mg	Standard deviation of correction, mg	Standard deviation for a set, mg
1	5 kg	•	66.624	7.06	5.87
2	2 kg	•X	25.649	3.69	5.87
3	2 kg	•	15.649	3.69	5.87
4	1 kg	•	16.039	2.30	2.89
5	500 g	•	10.698	1.06	1.54
6	200 g	•X	.279	1.25	1.54
7	200 g	•	.279	1.25	1.54
8	100 g	•	2.283	1.41	1.54
9	50 g	•	4.713	1.31	4.42
10	20 g	•X	.885	1.29	4.42
11	20 g	•	-4.115	1.29	4.42
12	10 g	•	.800	1.42	4.42
13	5 g	•	2.543	1.31	2.44
14	2 g	•X	.874	1.29	2.44
15	2 g	•	.160	1.29	2.44
16	1 g	•	-.634	1.42	2.44

CALIBRATION OF 5- TO 1-kg, CLASS 2 WEIGHTS

Calibration of the 5- to 1-kg, class 2 set of weights was accomplished using the procedure outlined under "Calibration Within a Set of Weights" and NIST design C.10.5.2.2.1.1.1 (appendix C).

Observations

$Y_{(1)}$ = 10 mg
$Y_{(2)}$ = 30 mg
$Y_{(3)}$ = 30 mg
$Y_{(4)}$ = 10 mg
$Y_{(5)}$ = 25 mg
$Y_{(6)}$ = 20 mg
$Y_{(7)}$ = 10 mg
$Y_{(8)}$ = 20 mg
$^{6}R_1$ = 11.039 mg

These observation values and R_1 were then used in equation 1 to calculate the true masses, which are

X_1 = 5 kg + 93.052 mg,

X_2 = 2 kg + 34.935 mg,

and X_3 = 2 kg + 18.507 mg,

The deviations were

Dev (1) = -2.86 mg
Dev (2) = 2.86 mg
Dev (3) = 0
Dev (4) = 0.71 mg
Dev (5) = -0.71 mg
Dev (6) = 2.14 mg
Dev (7) = 2.14 mg
Dev (8) = -3.57 mg

The sum of the deviations squared is 39.27, which divided by 3 degrees of freedom is 13.09. The square root of 13.09 is 3.62 mg, the standard deviation of this set of measurements.

[6] R_1 is found in the report of calibration for the class M set standard for 1-kg weight (table 1).

CALIBRATION OF 1-kg, CLASS 2 WEIGHTS

Calibration of the 1-kg, class 2 weight was accomplished using the procedure outlined under "Calibration of 1-kg Weights" and NIST design A.1.2 (appendix A).

Observations

$Y_{(1)}$ = 0
$Y_{(2)}$ = 0
$Y_{(3)}$ = 5 mg
$Y_{(4)}$ = 5 mg
$Y_{(5)}$ = 5 mg
$Y_{(6)}$ = 0
$^{7}R_1$ = 11.039 mg

These observation values and R_1 were then used in equation 1 to calculate the true masses, which are

X_1 = kg + 14.789 mg

and X_2 = kg + 16.039 mg.

R_2 = 16.039 mg, which will be the restraint used in the next weighing set.

The deviations were

Dev (1) = 1.25 mg
Dev (2) = -2.50 mg
Dev (3) = 1.25 mg
Dev (4) = 1.25 mg
Dev (5) = 0
Dev (6) = -1.25 mg

The sum of the deviations squared is 12.50, which divided by 3 degrees of freedom is 4.17. The square root of 4.17 is 2.04 mg, the standard deviation of this set of measurements.

[7] R_1 is found in the report of calibration for the class M set (table 1) and is the true mass correction value for the 1-kg weight.

CALIBRATION OF 500- TO 100-g, CLASS 2 WEIGHTS

Calibration of the 500- to 100-g, class 2 set of weights was accomplished using the procedure outlined under "Calibration Within a Set of Weights" and NIST design C.10.5.2.2.1.1.1 (appendix B).

Observations

$Y_{(1)}$ = 5 mg
$Y_{(2)}$ = 0 mg
$Y_{(3)}$ = 5 mg
$Y_{(4)}$ = 0 mg
$Y_{(5)}$ = 0 mg
$Y_{(6)}$ = 5 mg
$Y_{(7)}$ = 0 mg
$Y_{(8)}$ = 5 mg
$^{8}R_2$ = 16.039 mg

These observation values and R_2 are then used in equation 1 to calculate the true masses, which are

X_1 = 500 g + 9.091 mg,

X_2 = 200 g + 3.065 mg,

X_3 = 200 g + 0.208 mg,

X_4 = 100 g + 3.675 mg,

and X_5 = 100 g + 1.533 mg.

R_3 = 1.533 mg, which will be the restraint used in the next weighing set.

The deviations were

Dev (1) = 2.14 mg
Dev (2) = -1.43 mg
Dev (3) = -0.71 mg
Dev (4) = 1.43 mg
Dev (5) = -1.43 mg
Dev (6) = 0
Dev (7) = -1.43 mg
Dev (8) = 2.86 mg

The sum of the deviations squared is 21.44, which divided by 3 degrees of freedom is 7.15. The square root of 7.15 is 2.67 mg, the standard deviation of this set of measurements.

[8] R_2 is found in the calibration of the 1-kg, class 2 weight and is the true mass correction value for mass X_2.

CALIBRATION OF 50- TO 10-g, CLASS 2 WEIGHTS

Calibration of the 50- to 10-g, class 2 set of weights was accomplished using the procedure outlined under "Calibration Within a Set of Weights" and NIST design C.10.5.2.2.1.1.1 (appendix B).

Observations

$Y_{(1)}$ = 5 mg
$Y_{(2)}$ = 5 mg
$Y_{(3)}$ = 0
$Y_{(4)}$ = 0
$Y_{(5)}$ = 0
$Y_{(6)}$ = 0
$Y_{(7)}$ = 0
$Y_{(8)}$ = 5 mg
$^{9}R_3$ = 1.533 mg

These observation values and R_3 were then used in equation 1 to calculate the true masses, which are

X_1 = 50 g + 2.909 mg,

X_2 = 20 g + (-0.122) mg,

X_3 = 20 g + (-1.551) mg,

X_4 = 10 g + 0.296 mg,

and X_5 = 10 g + 2.439 mg.

R_4 = 2.439 mg, which will be the restraint used in the next weighing set.

The deviations were

Dev (1) = 2.14 mg
Dev (2) = -0.71 mg
Dev (3) = -1.43 mg
Dev (4) = 0.71 mg
Dev (5) = -0.71 mg
Dev (6) = 0.71 mg
Dev (7) = -2.14 mg
Dev (8) = 2.14 mg

The sum of the deviations squared is 17.80, which divided by 3 degrees of freedom is 5.93. The square root of 5.93 is 2.44 mg, the standard deviation of this set of measurements.

[9] R_3 is found in the calibration data of the 500- to 100-g, class 2 set and is the true mass correction value for mass X_5.

CALIBRATION OF 5- TO 1-g, CLASS 2 WEIGHTS

Calibration of the 5- to 1-g, class 2 set of weights was accomplished using the procedure outlined under "Calibration Within a Set of Weights" and NIST design C.10.5.2.2.1.1.1 (appendix B).

Observations

$Y_{(1)} = 0$
$Y_{(2)} = 0$
$Y_{(3)} = 0$
$Y_{(4)} = 0$
$Y_{(5)} = 0$
$Y_{(6)} = 0$
$Y_{(7)} = 5$ mg
$Y_{(8)} = 5$ mg
$^{10}R_4 = 2.439$ mg

These observation values and R_4 were then used in equation 1 to calculate the true masses, which are

$X_1 = 5$ g + 1.577 mg,

$X_2 = 2$ g + 2.059 mg,

$X_3 = 2$ g + (-0.798) mg,

and $X_4 = 1$ g + (-0.399) mg.

The deviations were

Dev (1) = 0 mg
Dev (2) = -1.43 mg
Dev (3) = 1.43 mg
Dev (4) = 1.43 mg
Dev (5) = -1.43 mg
Dev (6) = -1.43 mg
Dev (7) = 1.43 mg
Dev (8) = 1.43 mg

The sum of the deviations squared is 14.31, which divided by 3 degrees of freedom is 4.77. The square root of 4.77 is 2.18 mg, the standard deviation of this set of measurements.

[10] R_4 is found in the calibration data of the 50- to 10-g, class 2 set and is the true mass correction value for mass X_5.

CALIBRATION OF 5- TO 1-kg, CLASS 4 WEIGHTS

Calibration of the 5- to 1-kg, class 4 set of weights was accomplished using the procedure outlined under "Calibration of 1-kg Weights." Comparisons and calculation of correction values, deviations, and the standard deviation were made using NIST design C.10.5.2.2.1.1.1 (appendix C).

Observations

$Y_{(1)} = 20$ mg
$Y_{(2)} = 15$ mg
$Y_{(3)} = 10$ mg
$Y_{(4)} = 15$ mg
$Y_{(5)} = 15$ mg
$Y_{(6)} = 10$ mg
$Y_{(7)} = 10$ mg
$Y_{(8)} = 15$ mg
$^{11}R_1 = 11.039$ mg

These observation values and R_1 were then used in equation 1 to calculate the true masses, which are

$X_1 = 5$ kg + 66.624 mg,

$X_2 = 2$ kg + 25.649 mg,

and $X_3 = 2$ kg + 15.649 mg.

The deviations were

Dev (1) = 3.57 mg
Dev (2) = -1.43 mg
Dev (3) = -2.14 mg
Dev (4) = 5.00 mg
Dev (5) = -5.00 mg
Dev (6) = 2.14 mg
Dev (7) = -2.14 mg
Dev (8) = 5.00 mg

The sum of the deviations squared is 103.53, which divided by 3 degrees of freedom is 34.51. The square root of 34.51 is 5.87 mg, the standard deviation of this set of measurements.

[11] R_1 is found in the report of calibration for the class M set (table 1) and is the true mass correction value for the 1-kg weight.

CALIBRATION OF 1-kg, CLASS 4 WEIGHTS

For calibration of the 1-kg weight, the procedure outlined in the "Calibration of 1-kg Weights" section was used. NIST design A.1.2 (appendix A) was employed for calculation of the correction values, deviation values, and standard deviation.

Observations

$Y_{(1)}$ = 5 mg
$Y_{(2)}$ = 0
$Y_{(3)}$ = 5 mg
$Y_{(4)}$ = 0
$Y_{(5)}$ = 5 mg
$Y_{(6)}$ = 0
$^{12}R_1$ = 11.039 mg

These observation values and R_1 were then used in equation 1 to calculate the true masses, which are

X_1 = 1 kg + 16.039 mg,

and X_2 = 1 kg + 13.539 mg.

R_5 = 13.539 mg, which will be the restraint used in the next weighing set.

The deviations were

Dev (1) = 2.50 mg
Dev (2) = -2.50 mg
Dev (3) = 0
Dev (4) = 0
Dev (5) = 2.50 mg
Dev (6) = -2.50 mg

The sum of the deviations squared is 25.0, which divided by 3 degrees of freedom is 8.33. The square root of 8.33 is 2.89 mg, the standard deviation of this set of measurements.

[12] R_1 is found in the report of calibration for class M set (table 1) and is the true mass correction value for mass X_2.

CALIBRATION OF 500- TO 100-g, CLASS 4 WEIGHTS

Calibration of the 500- to 100-g, class 4 weights was accomplished by following the steps outlined under "Calibration Within a Set of Weights." Comparisons and calculation of the correction values, deviations values, and standard deviation were made using NIST design C.10.5.2.2.1.1.1 (appendix B).

Observations

$Y_{(1)}$ = 5 mg
$Y_{(2)}$ = 10 mg
$Y_{(3)}$ = 10 mg
$Y_{(4)}$ = 5 mg
$Y_{(5)}$ = 5 mg
$Y_{(6)}$ = 0 mg
$Y_{(7)}$ = 0 mg
$Y_{(8)}$ = 0 mg
$^{13}R_5$ = 13.539 mg

These observation values and R_5 were then used in equation 1 to calculate the true masses, which are

X_1 = 500 g + 10.698 mg,

X_2 = 200 g + 0.279 mg,

X_3 = 200 g + 0.279 mg,

X_4 = 100 g + 2.283 mg,

and X_5 = 100 g + 2.283 mg.

R_6 = 2.283 mg, which will be the restraint used in the next weighing set.

The deviations were

Dev (1) = -1.43 mg
Dev (2) = 0.71 mg
Dev (3) = 0.71 mg
Dev (4) = 0
Dev (5) = 0
Dev (6) = 0
Dev (7) = 1.43 mg
Dev (8) = -1.43 mg

The sum of the deviations squared is 7.14, which divided by 3 degrees of freedom is 2.38. The square root of 2.38 is 1.54 mg, the standard deviation of this set of measurements.

[13] R_5 is found in the report of calibration of the 1-kg, class 4 weight and is the true mass correction value for mass X_2.

CALIBRATION OF 50-to 10-g, CLASS 4 WEIGHTS

Calibration of the 50- to 10-g, class 4 set of weights was accomplished using the procedure outlined under "Calibration Within a Set of Weights." The comparison and calculation of correction values, deviations, and the standard deviation were made using NIST design C.10.5.2.2.1.1.1 (appendix B).

Observations

$Y_{(1)} = 5$ mg
$Y_{(2)} = 10$ mg
$Y_{(3)} = 5$ mg
$Y_{(4)} = 5$ mg
$Y_{(5)} = 0$
$Y_{(6)} = 5$ mg
$Y_{(7)} = 5$ mg
$Y_{(8)} = 10$ mg
$^{14}R_6 = 2.283$ mg

These observation values and R_6 were then used in equation 1 to calculate the true masses, which are

$X_1 = 50$ g + 4.713 mg,

$X_2 = 20$ g + 0.885 mg,

$X_3 = 20$ g + (-4.115) mg,

$X_4 = 10$ g + 0.800 mg,

and $X_5 = 10$ g + 2.943 mg.

$R_7 = 2.943$ mg, which will be the restraint used in the next weighing set.

The deviations were

Dev (1) = 0.71 mg
Dev (2) = -1.43 mg
Dev (3) = -0.71 mg
Dev (4) = 5.00 mg
Dev (5) = -5.00 mg
Dev (6) = 0.71 mg
Dev (7) = 0.71 mg
Dev (8) = 2.14 mg

The sum of the deviations squared is 58.67, which divided by 3 degrees of freedom is 19.56. The square root of 19.56 is 4.42 mg, the standard deviation of this set of measurements.

[14] R_6 is found in the calibration data of the 500- to 100-g, class 4 set and is the true mass correction value for mass X_5.

CALIBRATION OF 5- TO 1-g, CLASS 4 WEIGHTS

Calibration of the 5- to 1-g, class 4 set of weights employed the procedure outlined under "Calibration Within a Set of Weights." The comparisons and calculation of the correction values, deviations, and standard deviation were made using NIST design C.10.5.2.2.1.1.1 (appendix B).

Observations

$Y_{(1)} = 5$ mg
$Y_{(2)} = 0$
$Y_{(3)} = 0$
$Y_{(4)} = 0$
$Y_{(5)} = 5$ mg
$Y_{(6)} = 0$
$Y_{(7)} = 0$
$Y_{(8)} = 0$
$^{15}R_7 = 2.943$ mg

These observation values and R_7 were then used in equation 1 to calculate the true masses, which are

$X_1 = 5$ g + 2.543 mg,

$X_2 = 2$ g + 0.874 mg,

$X_3 = 2$ g + 0.160 mg,

and $X_4 = 1$ g + (-0.634) mg.

The deviations were

Dev (1) = 1.43 mg
Dev (2) = -0.71 mg
Dev (3) = -0.71 mg
Dev (4) = -2.14 mg
Dev (5) = 2.14 mg
Dev (6) = -0.71 mg
Dev (7) = -2.14 mg
Dev (8) = 0.71 mg

The sum of the deviations squared is 17.80, which divided by 3 degrees of freedom is 5.93. The square root of 5.93 is 2.44 mg, the standard deviation of this set of measurements.

[15] R_7 is found in the calibration data of the 50- to 10-g, class 4 set and is the true mass correction value for mass X_5.

DISCUSSION OF RESULTS

The data reported in tables 2 and 3 show the precision that can be achieved using the Voland model HCE 100G analytical balance as a comparator, when calibrating class 2 and class 4 sets of weights. The standard deviations of each set of weights were generally within ±1 mg of the ±2.91-mg balance sensitivity.

CONCLUSIONS

This method of calibration of standards of mass is adequate to obtain the true masses of weights that are used as counterbalances in determining the true masses of individual gases in primary gas standard preparation. A unique feature of this method is that weighing designs are employed that provide a self-consistency check of the calibration process.

The standard deviation for the class 2 weights, when compared to the NIST class M calibrated 1-kg weight, was determined for each set. The standard deviation was ±3.62 mg for the 5-kg weight set, ±2.67 mg for the 500-g weight set, ±2.44 mg for the 50-g weight set, and ±2.18 mg for the 1-g weight set.

The standard deviation for the class 4 weights, when compared to the NIST class M calibrated 1-kg weight, was determined for each set. The standard deviation was ±5.87 mg for the 5-kg weight set, ±1.54 mg for the 500-g weight set, ±4.42 mg for the 50-g weight set, and ±2.44 mg for the 1-g weight set.

APPENDIX A.—MULTIPLIERS OF THE OBSERVATIONS FOR DETERMINING THE CALCULATED CORRECTION VALUES AND DEVIATION VALUES FOR FOUR EQUAL WEIGHTS X_1 TO X_4, NIST DESIGN A.1.2

The restraint for this design is that weight x_4 has previously been calibrated and is used for the known standard.

Parameter values $[K_{(X_n,Y_n)}]$, * divisor = 4

Observations	X_1	X_2	X_3	X_4
Y_1	1	-1	0	0
Y_2	1	0	-1	0
Y_3	2	1	1	0
Y_4	0	1	-1	0
Y_5	1	2	1	0
Y_6	1	1	2	0
M	4	4	4	4

* Defined in equation 1.

where $K_{(X_n,Y_n)}$ = parameter values,

degrees of freedom = 3,

and $M_{(X_n)}$ = restraint values for the design.

Deviation parameter values $[L_{(Z_n,Y_n)}]$,** divisor = 4

Observations	Z_1	Z_2	Z_3	Z_4	Z_5	Z_6
Y_1	2	-1	-1	1	1	0
Y_2	-1	2	-1	-1	0	1
Y_3	-1	-1	2	0	-1	-1
Y_4	1	-1	0	2	-1	1
Y_5	1	0	-1	-1	2	-1
Y_6	0	1	-1	1	-1	2

** Defined in equation 2.

where $L_{(Z_n,Y_n)}$ = deviation values for the design.

APPENDIX B.—MULTIPLIERS OF THE OBSERVATIONS FOR DETERMINING THE CALCULATED CORRECTION VALUES AND DEVIATION VALUES FOR WEIGHTS X_1 TO X_6, LESS THAN 1 kg, NIST DESIGN C.10.5.2.2.1.1.1

The restraint for this design is that the sum of weights X_1, X_2, X_3, and X_4 has previously been calibrated and is used for the known standard.

Parameter values $[K_{(X_n,Y_n)}]$, * divisor = 70

Observations	X_1	X_2	X_3	X_4	X_5	X_6
Y_1	15	-8	-8	1	1	21
Y_2	15	-8	-8	1	21	1
Y_3	5	-12	-12	19	-1	-1
Y_4	0	2	12	-14	-14	-14
Y_5	0	12	2	-14	-14	-14
Y_6	-5	8	-12	9	-11	-1
Y_7	5	12	-8	-9	1	11
Y_8	0	10	-10	0	10	-10
M	35	14	14	7	7	7

* Defined in equation 1.

where $K_{(X_n,Y_n)}$ = parameter values,

degrees of freedom = 3,

and $M_{(X_n)}$ = restraint values for the design.

Deviation parameter values $[L_{(Z_n,Y_n)}]$,** divisor = 7

Observations	Z_1	Z_2	Z_3	Z_4	Z_5	Z_6	Z_7	Z_8
Y_1	2	-1	-1	0	0	0	-2	2
Y_2	-1	2	-1	0	0	2	0	-2
Y_3	-1	-1	2	0	0	-2	2	0
Y_4	0	0	0	3	-3	1	1	1
Y_5	0	0	0	-3	3	-1	-1	-1
Y_6	0	2	-2	1	-1	3	-1	-1
Y_7	-2	0	2	1	-1	-1	3	-1
Y_8	2	-2	0	1	-1	-1	-1	3

** Defined in equation 2.

where $L_{(Z_n,Y_n)}$ = deviation values for the design.

APPENDIX C.—MULTIPLIERS OF THE OBSERVATIONS FOR DETERMINING THE CALCULATED CORRECTION VALUES AND DEVIATIONS FOR THE CALIBRATION OF WEIGHTS X_1 TO X_6, LARGER THAN 1 kg, NIST DESIGN C.10.5.2.2.1.1.1

The restraint for this design is that weight X_6 has previously been calibrated and is used for the known standard.

Parameter values $[L_{(Z_n, Y_n)}]$, * divisor = 7

Observations	X_1	X_2	X_3	X_4	X_5	X_6
Y_1	-9	-5	-5	-2	-2	0
Y_2	1	-1	-1	0	2	0
Y_3	1	-1	-1	2	0	0
Y_4	7	3	4	0	0	0
Y_5	7	4	3	0	0	0
Y_6	0	1	-1	1	-1	0
Y_7	-5	-1	-3	-2	-1	0
Y_8	5	3	1	1	2	0
M	35	14	14	7	7	7

* Defined in equation 1.

where $K_{(X_n, Y_n)}$ = parameter values for the design,

degrees of freedom = 3,

and $M_{(X_n)}$ = the corresponding restraint values for the design.

Deviation parameter values $[L_{(Z_n, Y_n)}]$,** divisor = 7

Observations	Z_1	Z_2	Z_3	Z_4	Z_5	Z_6	Z_7	Z_8
Y_1	2	-1	-1	0	0	0	-2	2
Y_2	-1	2	-1	0	0	2	0	-2
Y_3	-1	-1	2	0	0	-2	2	0
Y_4	0	0	0	3	-3	1	1	1
Y_5	0	0	0	-3	3	-1	-1	-1
Y_6	0	2	-2	1	-1	3	-1	-1
Y_7	-2	0	2	1	-1	-1	3	-1
Y_8	2	-2	0	1	-1	-1	-1	3

** Defined in equation 2.

where $L_{(Z_n, Y_n)}$ = deviation values for the design.

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